

# DESIGN

**Project Title:** High Resolution Interfacial Microstructural Studies of Ceramic Materials (89-046)  
*J. Hefter and C. Sung*

User Center: MAUC

Status: Continuing

The development of advanced toughened silicon nitride composites requires understanding of the relationship between the intergranular second phase and the silicon nitride matrix.

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Metals and Ceramics Division

**HIGH TEMPERATURE MATERIALS LABORATORY  
THIRD  
ANNUAL REPORT  
(OCTOBER 1989 THROUGH SEPTEMBER 1990)**

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# HIGH TEMPERATURE MATERIALS LABORATORY THIRD ANNUAL REPORT\* (OCTOBER 1989 THROUGH SEPTEMBER 1990)

V. J. Tennery and F. M. Foust

## ABSTRACT

The High Temperature Materials Laboratory has completed its third year of operation as a designated DOE User Facility at the Oak Ridge National Laboratory. Growth of the user program is evidenced by the number of outside institutions who have executed user agreements since the facility began operation in 1987. A total of 88 nonproprietary agreements (40 university and 48 industry) and 20 proprietary agreements (1 university, 19 industry) are now in effect.

Sixty-eight nonproprietary research proposals (39 from university, 28 from industry, and 1 other government facility) and 8 proprietary proposals were considered during this reporting period. Research projects active in FY 1990 are summarized.

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## INTRODUCTION

The High Temperature Materials Laboratory (HTML) is a modern research facility which houses an array of special research equipment used to meet research needs for advanced high-temperature materials, including structural ceramics and alloys. The research instruments in the original four HTML User Centers provide a comprehensive set of tools for performing state-of-the-art determination of the structure and properties of solids. A key part of the HTML concept includes a staff of highly trained technical personnel who interact with industrial and university researchers in this Department of Energy (DOE) designated National User Facility. The User Centers are organized to provide materials characterization support to appropriate university and industrial users and to research programs throughout the local DOE facilities. Support includes a wide range of involvements with research personnel such as (1) conducting research relating materials properties to structure, (2) characterization of one-of-a-kind specimens, and (3) training qualified users and then providing them access to equipment to perform their own materials research.

User Agreements were developed which establish the intellectual property and liability rights of the user institution and the Oak Ridge National Laboratory (ORNL). The first official User Agreement was signed on July 15, 1987. Since that time, over 100 agreements have been executed. The first users from the university and industrial community started their research projects in August of 1987.

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Approval was received during this reporting period to start two new user centers: (1) X-Ray Residual Stress and (2) Ceramic Specimen Preparation. A more complete description of these new centers is included in Appendix A with the descriptions of the capabilities of the four original User Centers. A listing of the HTML User Center staff is also included in Appendix A.

## COMMITTEE ACTIVITIES

Three advisory committees assist in the successful operation of the HTML User Program. These committees are listed below with a brief description of their organization, function, and activities during the past year. A listing of members of these committees is included in Appendix B.

Advisory Committee. This senior committee has the responsibility of advising the HTML Director on policy for operation of the User Centers. It is composed of six members who represent the industrial and academic communities. The Committee meets at least annually and more frequently when its advice is urgently required on particular matters. A formal report containing conclusions resulting from a meeting of the Committee is submitted to the Associate Director of Physical Sciences of ORNL.

User Advisory Committee. The responsibility of this committee is to review nonproprietary research proposals and make recommendations to the HTML Director as to their acceptability. It is composed of six members - two from industry, one from a university, one from DOE, one from the M&C Division staff, and the HTML Director, who serves as the permanent chairman. A more detailed description of this committee's function and responsibilities is given in the brochure, User Program for the High Temperature Materials Laboratory. This committee reviewed 68 research proposals in FY 1990 (January, March, June, and August).

HTML User Exchange Group. This group was formed and a first meeting held in the HTML on August 18, 1989. A second meeting is in the planning stages. Present, past, and potential users of the HTML are invited to these meetings, which provide current and past users an opportunity to give advice on how the user program can be improved and made more effective.

## USER AGREEMENTS

There are two User Agreements utilized in the HTML User Program. The "Standard Nonproprietary Agreement" is for research projects whose results are reported in the open literature within six months of the completion of the project in the HTML. The "Standard Proprietary Agreement" is used for all projects in which the user desires that the data and results be proprietary. Table 1 is a listing of institutions who have executed nonproprietary user agreements to date.

## USERS

A cumulative summary of user activity since the start of the User Program is included as Appendix C.

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Table 1. Standard User Agreements executed July 15, 1987 through September 30, 1990

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**AGREEMENTS EXECUTED JULY 15, 1987 THROUGH SEPTEMBER 1988**

**UNIVERSITIES**

Alfred University  
Auburn University  
Clemson University  
Dartmouth College  
Georgia Institute of Technology  
New Mexico Institute of Mining  
and Technology  
North Carolina State University  
Oklahoma State University  
Pennsylvania State University

Southern Illinois University  
University of Alabama  
University of Illinois  
University of Michigan  
University of New Mexico  
University of Southern California  
University of Tennessee  
Vanderbilt University  
Virginia Polytechnic Institute  
and State University

**INDUSTRIES**

Allied Signal  
American Matrix, Inc.  
Ceramics Process Systems Corp.  
Dow Corning Corp./Midland

Great Lakes Research Corp.  
Norton Company  
Nuclear & Aerospace Materials Corp.  
Selee Corp.

**PROPRIETARY\* - 3 Agreements**

**AGREEMENTS EXECUTED OCTOBER 1988 THROUGH SEPTEMBER 1989**

**UNIVERSITIES**

Johns Hopkins University  
Marquette University  
Rice University  
Southern University  
University of California at  
Los Angeles

University of Denver  
University of Florida  
University of Minnesota  
University of Missouri-Rolla  
University of Utah

**INDUSTRIES**

Carborundum Company  
Coors Ceramics Company  
DG Trim Products  
Dow Chemical Company  
Energy Conversion Devices, Inc.  
Foster-Miller, Inc.  
GTE Laboratories, Inc.  
IMTech Company  
Institute for Defense Analysis

Ionic Atlanta, Inc.  
Litton Industries  
Sullivan Mining Corp.  
Tennessee Center for R&D  
Textron Specialty Materials  
Thermacore, Inc.  
Third Millennium Technologies, Inc.  
Universal Energy Systems, Inc.

Table 1. Continued

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**OTHER GOVERNMENT FACILITIES**

Albany Research Center (Bureau of Mines)  
 EG&G Mound  
 National Institute of Standards and Technology

**PROPRIETARY\* - 6 Agreements****AGREEMENTS EXECUTED OCTOBER 1989 THROUGH SEPTEMBER 1990****UNIVERSITIES**

Brown University  
 Illinois Institute of Technology  
 Kent State University  
 Lehigh University  
 Michigan Technology University  
 Rice University

Rutgers University  
 Tuskegee University  
 University of Arizona  
 University of Cincinnati  
 University of Delaware  
 University of South Carolina

**INDUSTRIES**

Allison Gas Turbine-Division of General Motors  
 Aluminum Co. of America  
 CarboMedics, Inc.  
 Certainteed Corporation  
 Cummins Engine Company  
 Detroit Diesel Corporation  
 Engelhard Corporation  
 Ford Motor Company  
 McDonald Douglas Missile Systems Company  
 Miniature Precision Bearings

Monarch Tile, Inc.  
 Norton/TRW Ceramics  
 Proctor & Gamble Company  
 Refractory Testing Associates  
 ReMaxCo Technologies, Inc.  
 SB&TD Business Systems  
 Sundstrand Power Systems-  
 Division, Sundstrand Corp.  
 Teledyne Allvac  
 United Technologies Corporation

**OTHER GOVERNMENT FACILITIES**

U.S. Bureau of Mines Tuscaloosa Research Center

**PROPRIETARY\* - 11 Agreements**

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\*Due to the sensitive nature of many of these research activities, the names of the user institutions for proprietary agreements are not listed.

The category of users and number of user days accumulated during this third year of HTML operation are shown in Table 2.

Table 2. HTML FY 1990 User Experience

Type of user	Number of		User days
	institutions	individuals	
Industry	30	53	956
University	19	40	607
Local Oak Ridge users	2	90	1758

Figure 1 shows the total number of user days per quarter during FY 1990. The industrial user days varied from about 209 to 271 during a particular quarter, university user days ranged from 122 to 175, and local user days ranged from 348 to 592. Figure 2 shows the number of user days for all industrial and university users in the HTML for each quarter of FY 1990.

A short summary of the research performed by nonproprietary users is given for each User organization. A listing of publications and presentations resulting from user research projects is given in Appendix D.

To date, 180 research proposals have been received in the User Program. The status of these is given in Table 3. Access clearances were initiated on 290 individual researchers who were listed as principal investigators on these proposals.

Table 3. Status of research proposals submitted July 1987 through September 1990

User Type	Research Proposals						
	Submitted	Approved	Rejected	Withdrawn	Active	Pending	Completed
Industry	70	57	0	11	25	10	24
University	92	86	1	12	55	2	22
Other Government	4	4	0	0	2	1	1
Total Nonproprietary	166	147	1	23	82	13	47
Proprietary	14	11	0	3	6	1	4
Grand Totals	180	158	1	26	88	14	51



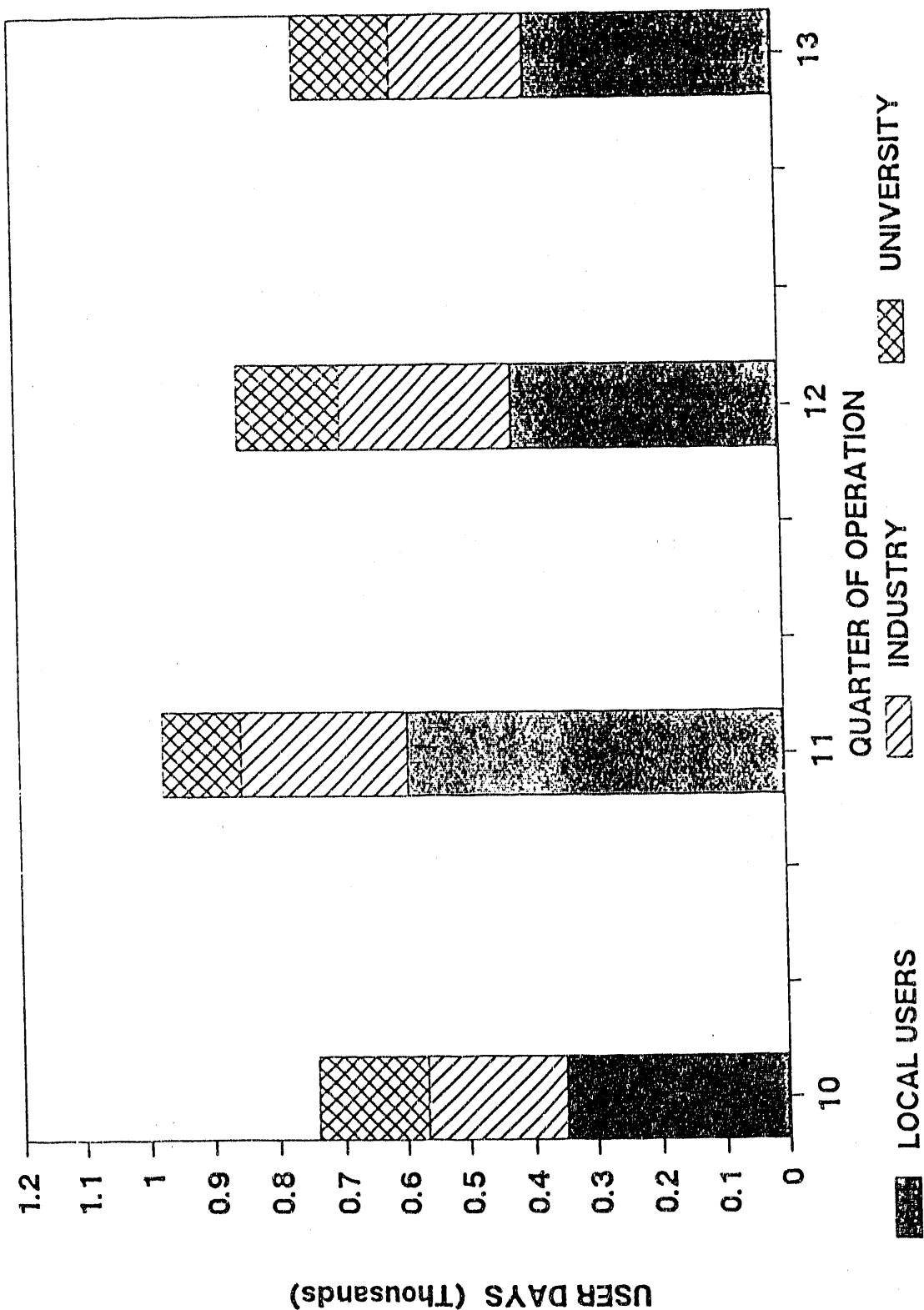


Fig. 1. Total HTML user days in FY 1990.

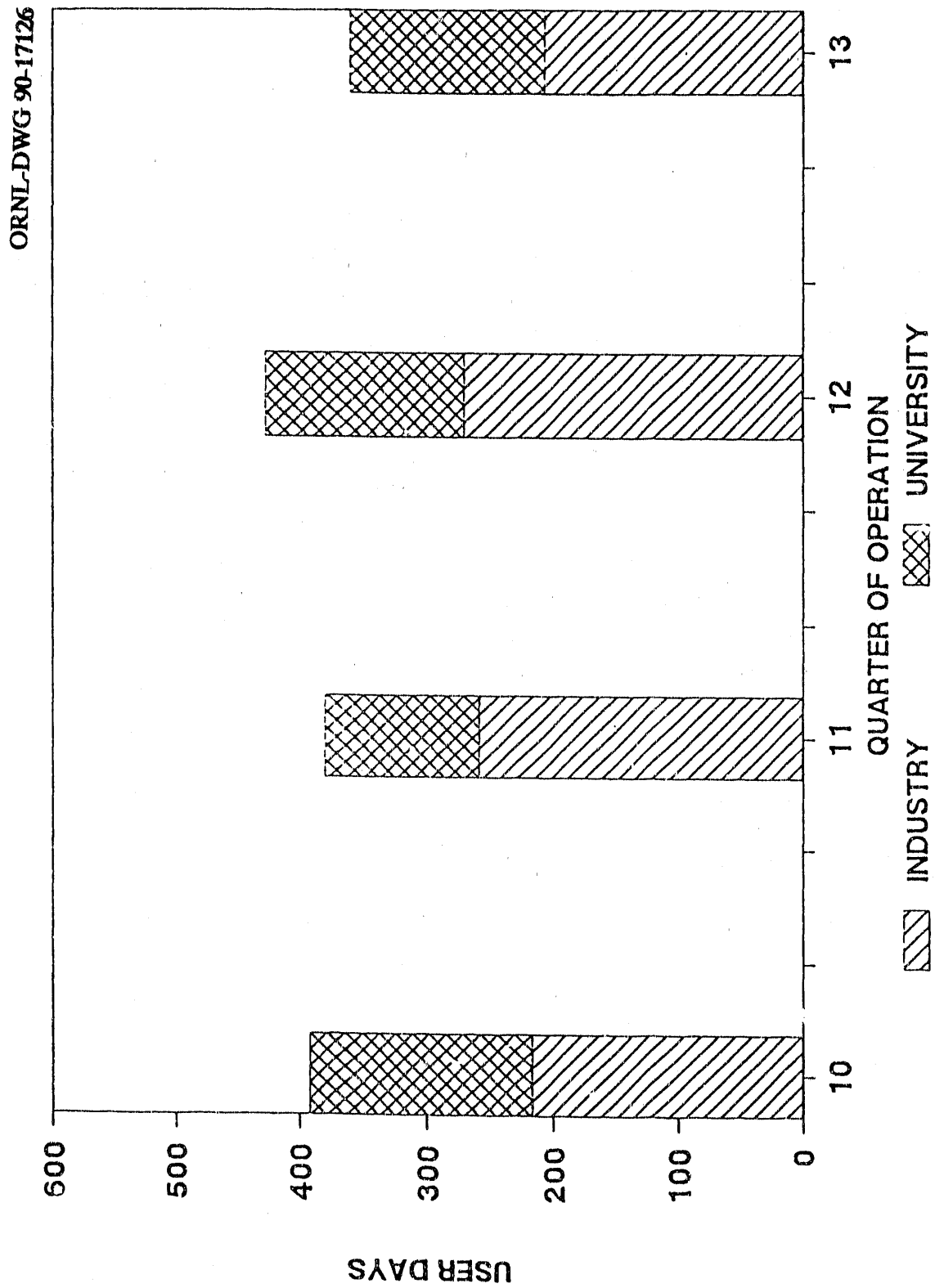


Fig. 2. Total industrial and university user days in the HTML in FY 1990.

## INDUSTRIAL USERS

A total of 53 researchers from 28 industrial firms and 2 government facilities spent a total of 956 days performing research on active proposals during FY 1990. The nonproprietary research projects which had significant activity during this period are briefly summarized.

### Aluminum Company of America

**Project Title:** Interfacial Properties Characterization in Nicalon SiC Fiber/Alumina-Based Composites (90-008) *H. Felix Wu*

User Center: MPUC

Status: Continuing

During the last visit to ORNL in July and August, measurements on our composites with the nanoindenter failed to debond uncoated or carbon-coated Nicalon SiC fibers in an alumina-based matrix. This might be due to the insufficient load capacity of the indenter or due to the improper sample preparation procedure. Currently, we are working to improve our metallographic sample preparation technique. We now plan to perform additional nanoindentation experiments in the HTML during October. We hope to be able to debond the coated fiber in the ceramic matrix composite sample to provide fiber shear strength and other properties critical to composite design and performance.

### Ceramics Process Systems Corporation

**Project Title:** Duophase Sialon with Controlled Grain Boundary Phase (89-052) *Ran-Rong Lee and D. L. Ouellette*

User Center: MPUC and XRDUC

Status: Continuing

Duophase sialons are currently under development at Ceramics Process Systems Corp. with assistance from the Cookson Company. The preliminary research results on the high-temperature four-point flexural strength at 1000, 1200, 1300, and 1370°C of these new duophase sialons are promising. This material can have strengths as high as 500 MPa at 1200°C and 490 MPa at 1370°C after optimum sintering and annealing. In comparison, the as-sintered material has only a 350 MPa flexural strength at 1300 and 1370°C. Transmission electron microscopy (TEM) and X-ray diffraction show that the crystallization of the grain boundary phase improved the high-temperature mechanical strength of our CM200 material. However, TEM characterization also revealed an inhomogeneous Y-Si-Al-N-O grain boundary phase, which is the factor believed to degrade high-temperature strength and high-temperature oxidation resistance. Further research on grain boundary phase engineering to improve high-temperature properties is proceeding.

**Cummins Engine Co., Inc.**

**Project Title:** Studies of Environmentally Induced Fracture of Diesel Engine Cylinder Head Cap Screws (90-010) *B. E. Wilde and J. E. Bokelman*

User Center: MAUC

Status: Continuing

This project was originated to determine the chemical nature of the species contributing to the environmentally induced cracking of Cummins cylinder head capscrews. Near the time that we were scheduled to start the work at HTML, other measurements done by Bryan Wilde and Jim Bokelman of Cummins indicated that temper embrittlement could be a factor influencing the failures. In discussions with staff of the Materials Analysis User Center plus staff at Cummins, it was decided to change the course of the project somewhat to try to determine the cause of the temper embrittlement. Auger examination of an initial sample from a failed capscrew fractured in situ revealed high levels of phosphorus grain boundary contamination. Efforts to confirm this hypothesis with other specimens has been unsuccessful to date, and the research is continuing.

**Dow Chemical Company**

**Project Title:** Determination of the Effect of Experimental Conditions on Lattice and Surface Oxygen Content of the AlN Phase (89-055) *G. Potter*

User Center: MAUC

Status: Continuing

The SIMS instrument was used to conduct measurements, both in the scanning and microprobe modes, to obtain images and spot analyses of AlN. This research is to determine the effect of experimental conditions including sintering agents on the lattice and surface oxygen content of the AlN phase and any second phase present in prepared AlN. The data collected in the HTML are being correlated with the thermal conductivity of the materials to determine the dependence of material performance on both types of oxygen measured. Enough samples were studied to allow quantification of both surface and lattice oxygen in these materials.

**EG&G Mound Laboratories**

**Project Title:** TEM Analysis of Metals in a Lithia-Alumina-Silica Glass Ceramic (89-043) *W. E. Moddeman*

User Center: MAUC

Status: Continuing

In this study, "zerovalent" nickel in lithium aluminosilicate (LAS) glass was characterized with high resolution XPS and with both analytical and ultrahigh resolution TEM. These results were presented at the 1990 American Ceramic Society Meeting (a poster won 2nd prize in its category) and at a Gordon Conference on Glass.

**Engelhard Corporation**

**Project Title:** Evaluation of the Thermal and Hydrothermal Stability of Fluid Cracking Catalyst Materials using High Temperature X-Ray Diffraction Analysis (90-016) *J. R. St. Amand*

User Center: XRDUC

Status: Completed

As fcc catalysts age in the field, aluminum is removed from the zeolite framework, the unit cell contracts and the selectivity of the catalysts is adversely affected. As a result, the amount of non-framework aluminum and Lewis acid sites increases. This in turn increases the amount of coking. Consequently, both changes in phase composition and unit cell size were monitored on three fcc catalyst samples to investigate the degradation of the zeolite component as a function of temperature.

**Great Lakes Research**

**Project Title:** Microstructural Examination of Polished  $\text{TiB}_2$ -Graphite Samples (00-003)

*E. G. Morris*

User Center: MAUC

Status: Continuing

Microprobe analyses were performed to determine the dissolution and migration of  $\text{TiB}_2$  and aluminum/cryolite penetration into a  $\text{TiB}_2$ -graphite composite exposed in an operating commercial hall process aluminum cell for up to 15 months. The objective of this work is to understand  $\text{TiB}_2$  degradation mechanisms in order to develop high reliability cathodes for aluminum reduction.

**GTE Laboratories, Inc.**

**Project Title:** Fracture Behavior of PY6 Silicon Nitride Specimens in Tension (89-050)

*A. E. Pasto and W. C. VanSchalkwyk*

User Center: MPUC

Status: Completed

Tensile strength measurements on a prototype set of ceramic tensile specimens at the HTML resulted in a large number of buttonhead failures, which were traced to an improper value of the buttonhead radius (3.0 mm radii specified, versus 2.5 mm radii provided by the machining laboratories). With these results, we had specimens prepared with a corrected radius, which resulted in a near-zero incidence of buttonhead failures in post-HTML measurements.

**Project Title:** High Resolution Interfacial Microstructural Studies of Ceramic Materials (89-046)  
*J. Hefter and C. Sung*

User Center: MAUC

Status: Continuing

The development of advanced toughened silicon nitride composites requires understanding of the relationship between the intergranular second phase and the silicon nitride matrix. Ultrahigh resolution microscopy was found to provide crucial information about the crystallinity and morphology of these phases, specifically when the composites were heat-treated to promote devitrification of the glassy phase. Atomic scale images of the interfaces between the silicon nitride matrix and the intergranular phase as well as images of the crystallized phases, their twin-like structure as well as other important crystallographic defects were provided and are being related to mechanical property data.

#### **Institute for Defense Analyses**

**Project Title:** High Temperature Testing of Ceramic Fibers (89-011) *W. S. Hong, J. M. Sater and M. A. Rigdon*

User Center: MPUC

Status: Completed

The purpose of this research was to systematically assess the effects of varying certain test parameters such as strain rate and thermal history on the measured high-temperature tensile strength and elastic modulus of a specific ceramic fiber, SCS-6, which has been used by many researchers for reinforcing ceramic and metal matrix composites. We have chosen the SCS-6 silicon carbide fiber from Textron Specialty Materials, and used the unique hot fiber strength measuring capability recently developed at the HTML. Fiber strength measurements were made at 1000°C and 1200°C, with two pretest soak times at temperature (3 min and 30 min) and two strain rates (0.2%/min and 2.0%/min, based on a gripped gage length of 155 mm). Initial results indicate that, for these fibers, changing the strain rate at 1200°C has had virtually no effect on ultimate tensile strength. At 1000°C, the measured average strengths increase as expected when using faster strain rates, indicating perhaps that a stress corrosion process was operative. At both temperatures, lower strengths were observed with increased soak times.

#### **McDonnell Douglas**

**Project Title:** Effects of Processing Conditions on the Thermophysical Properties of Carbon-Carbon Composites (90-092) *J. W. Sapp and D. A. Bowers*

User Center: PPUC

Status: Continuing

Utilizing the instruments in the Physical Properties User Center, data were collected to correlate the effects of processing conditions on the thermophysical properties of carbon-carbon composites. Four different carbon-carbon composite samples were used in measurements in an inert atmosphere to determine the following: thermal diffusivity, 200-1700°C; thermal expansion, 20-1450°C; and specific heat, 200-1200°C.

**Thermacore, Inc.**

**Project Title:** Refractory Metal Heat Pipe Compatibility with Lithium at Elevated Temperatures (89-007) *J. R. Hartenstine*

User Center: MAUC

Status: Completed

The purpose of the research was to evaluate refractory metal heat pipe compatibility with lithium at elevated temperatures. A series of tantalum heat pipes were fabricated using lithium as the working fluid. These heat pipes were operated at 1150°C for 1000 h. The heat pipes were subsequently sectioned and the tantalum envelope material was analyzed in the HTML user centers.

The analyses were performed utilizing the JEOL 733 electron microprobe. No significant corrosion of the tantalum envelope material was evident during the inspection of the evaporator and condenser sections. The results from the HTML effort indicated that the procedures and processes used to fabricate the tantalum/lithium heat pipes is capable of producing very long life refractory metal heat pipe.

**United Technologies Research Center**

**Project Title:** Effects of Aging Time and Temperature on the Thermal Conductivity of Plasma Sprayed Zirconia and Mullite for Thermal Barrier Coating Applications (90-009) *J. R. VanValzah* and *H. E. Eaton*

User Center: PPUC

Status: Continuing

The thermal conductivities of 30 plasma-sprayed zirconia and three mullite specimens were measured in the as-sprayed and thermally aged conditions. The zirconia aging matrix consisted of isothermal exposures at temperatures between 870°C and 1500°C for times ranging from 1 to 500 h. Conductivity was determined for each specimen over a range of temperatures between 200 and 1300°C. In addition, the effects of porosity type and level in the zirconia were examined. Mullite aging conditions were 870°C/100 h and 540°C/500 h. Results obtained to date show large increases in thermal conductivity due to aging with aging temperature being much more significant than aging time. Additionally, increasing the porosity level is, as expected, associated with a reduction in the thermal conductivity.

**Universal Energy Systems, Inc.**

**Project Title:** Characterization of the Oxide/Corrosion Scales on Three Classes of Iron Based Alloys (89-041) *V. Srinivasan*

User Center: MAUC and MPUC

Status: Completed

Oxide/corrosion scales were characterized at the HTML user facility using the scanning Auger spectrometer (SAM), electron spectroscopy for chemical analysis/secondary ion mass spectroscopy (ESCA/SIMS) and the nanoindenter. Corrosion scales were thermally grown under different exposure conditions that were typical of coal gasification/combustion. The experimental alloys were high purity austenitic and ferritic steels with and without microconstituent additions. The results indicate factors that enhance the protectiveness of thermally grown chromia scales in the presence of sulfur. Protective scales, according to the results of nanoindenter experiments, have lower stiffness (lower Young's modulus). This research is directed to advanced coal conversion systems.

**Project Title:** Study of Surface Modification and Coatings of SiC and Si<sub>3</sub>N<sub>4</sub> at High Temperatures (89-018) *R. S. Bhattacharya*

User Center: MPUC

Status: Continuing

The research involved co-implantation of Ti<sup>+</sup> and C<sup>+</sup>, and Cr<sup>+</sup> and C<sup>+</sup> into sintered SiC and hot-pressed Si<sub>3</sub>N<sub>4</sub>. Diffusion of dopants, recrystallization of the implanted layers, and formation of second phases upon annealing at high temperature were investigated. The purpose is to determine if precipitates of TiC and Cr<sub>3</sub>C<sub>2</sub> could be formed in the near surface region of SiC and Si<sub>3</sub>N<sub>4</sub>, and if the precipitate formation can help improve the surface hardness and toughness of these structural ceramics.

**U. S. Bureau of Mines**

**Project Title:** Microstructure and Chemistry of Specific Regions of a Fine Grain SiC Specimen Subjected to Laser Irradiation (90-011) *L. Y. Sadler*

User Center: MAUC

Status: Continuing

Fracture surfaces were studied using both the scanning electron microscope and optical microscopy. Excellent quality micrographs were taken of various regions of SiC powder compacts representing processing temperatures ranging from room temperature to in excess of 2000°C, and analyses of these structures are continuing.



## UNIVERSITY USERS

A total of 40 researchers from 19 universities spent a total of 607 days performing research on 43 active proposals. A brief summary of each nonproprietary proposal which had significant activity during this report period is given below.

### Alfred University

**Project Title:** Establishment of the Peritectic Reaction Sequence for the Bi-Sr-Ca-Cu-O Superconductors (90-032) *D. P. Matheis and R. L. Snyder*

User Center: PPUC and XRDUC

Status: Continuing

Research to date on this project in the HTML included high-temperature X-ray diffraction analysis as well as differential scanning calorimetry (DSC) analysis of melt-quenched samples of stoichiometric and non-stoichiometric  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  powders. The analysis is aimed at investigating the crystallization and peritectic melting reactions of these candidate superconducting materials.

### Clemson University

**Project Title:** Study of the Character of Stresses Developed in an Iron Based Alloy Subject to Sulfidizing Mixed Gases at Elevated Temperatures (87-013) *J. S. Wolf*

User Center: XRDUC

Status: Continuing

The primary objective of this research has been to provide some means for estimating the protective quality of scales formed in various aggressive atmospheres of interest at elevated temperatures. The crystalline nature of these scales, their microstructural evolution, and the stresses generated during corrosion were studied. One purpose of this research is to understand how the oxidation and corrosion resistance of the steels could be improved. For these tasks, much of the data collection and processing was done utilizing the X-ray Diffraction Facility of the HTML.

**Project Title:** Stress Generation In and Structure Of Scales Developed on Unalloyed Nickel (88-030) *J. S. Wolf*

User Center: XRDUC

Status: Continuing

This research has been directed toward the characterization of ceramic oxide scales naturally formed upon unalloyed nickel in oxygen at high temperatures. The primary objective of this research has been to investigate methods for estimating the growth stresses which occur in such oxides. The crystalline nature of these scales and the stresses generated during oxidation were the primary topics of investigation. For these tasks, much of the data collection and processing was done utilizing the high-temperature X-ray diffraction facility of the HTML.

**Project Title:** High Temperature X-Ray Analysis of Titanium Aluminides (88-034) *H. J. Rack, P. K. Chaudhury and M. G. Long*

User Center: XRUC

Status: Continuing

High-temperature phase stability for Ti-Al-Nb (Ti-24Al-11Nb and Ti-25Al-10Nb-3V-1Mo) and gamma-Ti-Al-V alloys has been completed. The results are presently being correlated with DTA/DSC and transmission electron microscopy efforts under way at Clemson for publication.

**Georgia Institute of Technology**

**Project Title:** Study of AlN-BN Composites (88-031) *J. A. Hanigofsky*

User Center: MAUC

Status: Completed

Completed transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) analysis on seven specimens of AlN + BN coatings deposited on Al<sub>2</sub>O<sub>3</sub>. The measurement and analysis time included about 3 weeks actual time at the HTML. Microstructural and elemental analyses were used to identify interfacial structures and elemental interdiffusion between the major phases.

**Project Title:** Moire Interferometry on Specimens of Lucalox Alumina Ceramics to Directly Measure Microstrains in Specimens (89-025) *J. S. Epstein and G. B. May*

User Center: MPUC

Status: Continuing

Extensive consultation has taken place between J. S. Epstein of Georgia Tech and M. Jenkins of the HTML on conversion of one of the Instron tension systems from strain to displacement control as well as selection of the correct specimen geometry and institutional requirements for fabricating the required Lucalox tension specimens. Preparation of the required specimens has been initiated.

**Project Title:** Study of the Concentration Profile in a Diffusion Couple after a Diffusion Anneal (90-022) *T. H. Sanders and H. R. Last*

User Center: MAUC

Status: Continuing

Electron microprobe measurements have been used to determine the composition profile in several diffusion-annealed couples. The diffusion couples investigated contain an Al-Cu or an Al-Mg alloy coupled with either an Al-Zr or an Al-Mn alloy. This information on the development of the concentration profile is being used with further diffusion experiments for the determination of diffusion parameters such as the activation energy for diffusion and the pre-exponential constant  $D_0$  for selected alloying elements. Also, this information will be used in conjunction with results obtained from annealing experiments performed on the couples to study precipitation as a function of alloy composition.

**North Carolina State University****Project Title:** Hard Carbon Coating on Silicon (90-014) *J. Park*

User Center: MPUC

Status: Continuing

The objective of this project is to investigate the hardness of diamondlike-carbon (DLC) films on silicon obtained by the laser ablation and deposition technique. Microhardness measurements have been performed on the samples using the nanoindenter. The samples were prepared with different substrate temperatures or with hydrogen content. More extensive experimental work is planned on hydrogenated DLC films.

**New Mexico Institute of Technology****Project Title:** Effect of Tin Dioxide Interlayer in Alumina/Glass Composites (88-029)  
*K. K. Chawla*

User Center: MAUC and MPUC

Status: Continuing

The objective of this project is to investigate the interface characteristics in composites in the system alumina/glass. Specifically, we wish to tailor the interface to obtain an enhanced fracture toughness. The alumina/glass system forms a very strong chemical bond, resulting in low fracture toughness. The interface characteristics can be changed by incorporating an interphase layer of tin dioxide between alumina and glass. The interphase layer acts as a diffusion barrier between the alumina and the glass, thereby inhibiting the chemical reaction and providing a weak layer and conditions highly favorable for fiber pullout, thus contributing to a high fracture toughness.

The work at HTML involves nanoindenter studies on alumina/glass and alumina/tin dioxide/glass systems, transmission electron microscopy (TEM) and secondary ion mass spectroscopy (SIMS) characterization, and high-temperature flexure strength measurements of these composites.

**University of California at Los Angeles****Project Title:** XPS Study of Quartz Dissolution Surfaces (89-062) *A. Gratz*

User Center: MAUC

Status: Completed

Quartz etched in hydroxide solutions were studied by X-ray photoelectron spectroscopy to determine the extent of cation binding to and the effect of dissolution on the surface. The surface was found to consist of a precipitated layer of adventitious carbon, cations from solution, and Si and O, deposited on an SiO<sub>2</sub> substrate. Cations above the substrate were readily exchanged and not concentrated at the substrate-deposit interface. We conclude that cations did not bind strongly to the substrate. Shifts in binding energy of O and Si were limited to the deposited layer, indicating poly ordered coating and an undisturbed substrate.

University of Denver

**Project Title:** Residual Stresses and Thermal Cycling in Ceramic Composites (89-030)  
*B. L. Ballard and P. K. Predecki*

User Center: XRDUC

Status: Continuing

The goal of this research project is to characterize the residual stress states found in a ceramic matrix composite consisting of alumina reinforced with silicon carbide whiskers ( $\text{Al}_2\text{O}_3/25 \text{ wt\% } \beta\text{-SiC}_w$ ) composite at elevated temperatures. Measurements using the HTML's X-ray diffractometer with its accompanying specimen heating furnace were used to characterize Bragg angle displacements in both phases of the specimen over a 25-1250°C temperature range. Also, values for the lattice thermal expansion of  $\beta\text{-SiC}_w$  over these temperatures were obtained during the course of the research. Results from the measurements are being analyzed.

University of Florida

**Project Title:** Characterization and Testing of Ceramic Oxide Fibers (89-009) *F. Feller, III*  
*A. A. Morrone and S. M. Sim*

User Center: MAUC and MPUC

Status: Continuing

Short, nonuniform fibers of  $\text{ZrO}_2/\text{Al}_2\text{O}_3$  and mullite have been synthesized. These have been analyzed at the HTML using the JEOL 2000FX analytical transmission electron microscope. We are developing continuous and uniform fibers of the same compositions. When these fibers are prepared, we plan to perform direct strength measurements on these fibers in the unique fiber strength facility in the HTML.

University of Illinois

**Project Title:** Effects of High Intensity Laser-Pulse-Generated Shock Waves on the Microstructures and Mechanical/Physical Properties of Metals (89-069) *J. M. Rigsbee and J. P. Chu*

User Center: MPUC and PPUC

Status: Continuing

The nanoindenter in the Mechanical Properties User Center was used to analyze the strength and deformation characteristics as a function of depth away from the shocked surface of materials. The materials studied were single-phase ferritic (pure iron - polycrystalline and single crystals) and austenitic (types 304L and 316 stainless steel) iron-based alloys. Data obtained to date are being analyzed and look very promising.

**Project Title:** Analysis of Modified Fibers and Composites in Nicalon SiC Fiber/MDF Cement-Polymer System (88-038) *D. M. Hansen and A. J. Burreson*

User Center: MPUC

Status: Completed

The nanoindenter was used to perform indentation tests on Nicalon SiC fiber reinforced calcium aluminate DSP cement composites. These tests were to examine the effects of different fiber surface treatments on the interfacial shear strength.

University of Michigan

**Project Title:** Study of Sintered Silicon Nitride/Silicon Carbide Composites and the Interface Structure Between the Silicon Nitride Matrix and the Silicon Carbide Whiskers (87-006)  
*T-Y. Tien and S. D. Nunn*

User Center: MAUC

Status: Completed

Microstructural evaluation of SiC whisker reinforced  $\text{Si}_3\text{N}_4$  composites was conducted using the JEOL 2000FX transmission electron microscope. The composition and morphology of the matrix, the whiskers, and the sintering aid additive which forms an interfacial layer at the grain boundaries was evaluated. Energy dispersive spectroscopy (EDS) analysis showed solid solution formation in the matrix ( $\beta'$ - $\text{Si}_3\text{N}_4$ ) and the intergranular phase (yttrium aluminum garnet). Crystallization of the grain boundary phase was confirmed by selected area diffraction. An unexpected finding was the observation of amorphous particles of the sintering aid material within  $\text{Si}_3\text{N}_4$  grains, apparently entrapped during crystallization of the  $\beta\text{Si}_3\text{N}_4$  grains from the liquid.

University of New Mexico

**Project Title:** Structure and Reactivity of Metal Particles supported on Model Oxide Surfaces (88-002) *A. K. Datye*

User Center: MAUC

Status: Continuing

This research is focused on understanding the interfacial structure between metals and oxide superconductors on the atomic level. The nature of Ag/1-2-3 superconductor interfaces in powders grown by aerosol decomposition was examined. In the future, we expect to devote more time to the analysis and simulation of high resolution transmission electron microscopy images derived from metal/ceramic interfaces from these model systems.

**Project Title:** Characterization of Polymer-Derived Ceramic Materials (88-003) *A. K. Datye*

User Center: MAUC

Status: Continuing:

The atomic structure of the BN/MgO interface has been successfully imaged. The results show that the interplanar spacing in the BN layers close to the interface is affected by the proximity of the MgO. The spacing between the BN sheets and MgO was found to be 3.5 Å suggesting either Van der Waal's type interaction or a bridging oxygen that serves to bind these two ceramic materials. Such information is vital to understanding the origins of macroscopic phenomena such as interfacial strength at the atomic scale.

During this reporting period, we also completed a study of the oxidative stability of BN coatings on MgO. While BN is stable up to 600°C in air, at higher temperature it is oxidized and reacts with MgO to form  $\text{Mg}_2\text{B}_2\text{O}_5$ .

University of Tennessee

**Project Title:** Study of Alumina/Metal and Metal/Molybdenum Carbide Interfaces Prepared via Excimer Laser and Sputter Deposition Techniques (87-016) *A. J. Pedraza and M. J. Godbole*

User Center: MA UC

Status: Continuing

The interface of a copper-sapphire couple that was irradiated with a nanosecond pulsed-excimer laser was studied by transmission electron microscopy. Deposited layers of 30 or 100 nm thickness were laser treated in an argon-4 vol % hydrogen atmosphere with energy densities in the range of 0.5-0.75 J/cm<sup>2</sup>. The copper film and a thin alumina layer were melted by the laser pulse. Two well-differentiated regions could be observed in the modified layer. The region closer to the unmodified substrate consisted of epitaxially regrown alumina with crystallites misoriented up to 10° relative to the substrate sapphire orientation, while precipitate particles could be seen closer to the resolidified copper. The precipitate compound exhibited a hexagonal structure closely related to sapphire where copper substituted for some aluminum. These observations are in agreement with a previously developed mathematical model that predicts melting of a thin substrate layer. These results strongly suggest that the observed three- to four-fold increase in adhesion in copper-sapphire couples is due to the presence of an intermediate compound.

VPI & SU

**Project Title:** Characterization of Bulk Hydrogen Concentrations and Hydrogen Concentration Profiles in Aluminum-Lithium Alloys (90-012) *R. E. Swanson and F. C. Rivet*

User Center: MAUC

Status: Completed

Various electrochemical charging techniques were used to produce internal hydrogen in several Al-Li alloys. The goal was to evaluate the role of hydrogen in the plastic deformation and low temperature mechanical behavior of these alloys. It was desired to produce internal hydrogen, at known levels, without inducing surface damage, which would lead to stress risers and spurious mechanical test results. The SIMS instrument was used to provide semi-quantitative results which showed that charging with either of the two methods listed below provided sufficient hydrogen. In support of this work, a noncontact optical profilometer showed that these charging methods produced essentially no surface damage.

Galvanostatic in 0.04 N HCl + As<sub>2</sub>O<sub>3</sub> in deionized water at -500 microA for 20 h  
Potentiostatic in same solution at -3V for 5 h

These results are significant in that they demonstrate that internal hydrogen can indeed be produced in these alloys without surface damage other investigators have experienced.

**Project Title:** Measurement of Boron Content of Intergranular Fracture Facets of a Boron-Doped Ni<sub>3</sub>Al Alloy (90-001) *Y-C. Lin*

User Center: MAUC

Status: Continuing

A sample of boron-doped Ni<sub>3</sub>Al having the configuration for fracture in the Auger spectrometer in the Materials Analysis User Center was prepared. The surfaces were metallographically polished and etched, and electron channeling patterns from about 40 grains in the vicinity of the notch, across which fracture would occur, were obtained using a scanning electron microscope at the University of Tennessee. This sample was then hydrogen charged to enhance grain boundary fracture, and immediately placed in the Auger spectrometer, and fractured within about 30 min. The boron content of about 20 grain boundary fracture facets at the edges for which channeling patterns were obtained was measured. We are now determining the relative misorientation of the pairs of grains through which the fracture propagated and this will be correlated with the B content.

**Project Title:** Examination of the Chemistry of the Microstructures of Weld Samples of Hastelloy B2 (90-020) *C. R. Brooks*

User Center: MAUC

Status: Continuing

Welds of the Ni-Mo alloy Hastelloy B2 in components of a coal gasification plant have shown intergranular corrosion. We are in the process of examining samples of these welds with secondary ion mass spectroscopy (SIMS) in the HTML to identify the fine grain boundary particles. The work is still in an early stage.

**Project Title:** Analysis of the Fracture Surfaces of Samples of a High-Temperature Flange Bolt Material (89-024) *C. R. Brooks*

User Center: MAUC

Status: Continuing

A metallographic sample of a 12% Cr embrittled flange bolt has been examined with secondary ion mass spectroscopy (SIMS) in the HTML. Particles rich in carbon and chromium have been identified in the prior austenite grain boundaries. Fracture surfaces are being examined.

**Project Title:** Study of the Fracture Surfaces of Ni-20 at. % Mo Alloys Which Have Been Microalloyed with Various Elements (89-023) *C. R. Brooks*

User Center: MAUC

Status: Continuing

A Ni-20 at. % Mo alloy containing a microalloying level of boron was prepared. A sample of this alloy was solution-annealed, quenched to retain the alpha phase, then aged for 1 h at 775°C. This treatment causes ordering and strengthening, but normally the alloy is brittle, fracturing along the former alpha phase high angle grain boundaries. The microalloying was an attempt to ductilize the alloy in the ordered condition, and the sample showed some ductility in a standard bend test. A sample of this alloy was fractured in the Auger spectrometer, and the fracture surfaces analyzed for boron. None was detected, indicating that essentially no boron

segregation occurred at the grain boundaries as a result of the thermal treatment. In this case, it appears that boron segregation to the grain boundaries is not the cause of the observed ductility. A paper on this work is being prepared.

**Project Title:** Research in Support of a Laser Welding Study on Inconel 718 (89-017)  
*N. B. Dahotre, M. H. McCay and T. D. McCay*

User Center: PPUC

Status: Completed

To accurately model the effects of laser pulse welding on crack sensitivity, certain physical properties of the high-temperature alloy Inconel 718 are required. Among these are the heating and cooling transformation temperatures for liquid and solid phases. Differential scanning calorimetry (DSC) and scanning thermal analysis (STA) were used to determine temperature zones for different transformations in wrought as well as cast Inconel 718. The samples were subjected to heating and cooling cycles with rates of 10 and 40°C/min in the temperature range 40-1350°C. The solidus temperature is about 1200°C and the alloy becomes completely liquid at about 1275°C. At about 1170°C a high-temperature phase formation occurs by solid state transformation which is not observed during cooling. A significant deviation in the base line slope by a factor of two in the DSC analysis in the temperature range 570-870°C indicated the presence of low-temperature phases.

**Project Title:** Study of Interfacial Reaction Phases in Laser Welded Ceramic Particulate/Al-alloy Metal Matrix Composite (89-056) *N. B. Dahotre and S. C. Gopinathan*

User Center: MAUC and XRDUC

Status: Continuing

The microstructural changes in the laser processed A356 Al alloy matrix composites reinforced with 10 and 20 vol % SiC particulates were characterized. The microstructure of the laser melted region was investigated using transmission electron microscopy and X-ray microchemical analysis techniques. Laser processing produced partial or complete dissolution of SiC particles and sometimes resulted in the formation of aluminum carbide. The associated rapid cooling also produced a fine distribution of nonequilibrium complex precipitates. In addition, the laser energy modified the SiC surface both physically and chemically. This research is continuing.

**Project Title:** Analysis of High Temperature Phase Transformation in Inconel 718 (89-057)  
*N. B. Dahotre, M. H. McCay and T. D. McCay*

User Center: XRDUC

Status: Completed

X-ray diffractometry data were obtained on Inconel 718 in the form of as-received cast, as-received wrought, and grain-growth wrought samples at room temperature using the HTML X-ray diffraction instruments. The X-ray diffraction data were also collected from the same samples at discrete temperatures (for as-received: 530, 580, 620, 720, 770 and 820°C; for as-received wrought: 540, 570, 600, 730, 760°C; and for wrought grain-grown: 590, 630, 730, 770, 800°C) by scanning them through 20 to 120° at 0.5°/min. In order to avoid surface oxidation effects, the high-temperature furnace was filled with argon during these experiments. Following the high-temperature runs, the room-temperature X-ray scans were run on the same sample to enable detection of irreversible transformation products as well as the disappearance of the reversible transformation products.



**Vanderbilt University****Project Title:** Wear Behavior of Cu-Al Solid Solution Alloys (89-047) *R. A. Poggie and J. J. Wert*

User Center: MAUC

Status: Continuing

The wear properties of Cu-Al alloys and the chemical bonding of selected elements on wear surfaces are major objectives of this research. A large body of data has been collected using x-ray photoelectron spectroscopy analyses of three copper-aluminum solid solution alloys (1, 4, and 6 wt % Al in Cu). The Cu-Al alloys surfaces analyzed to date were in the as-polished condition, ion-sputtered condition, and surfaces oxidized at ambient temperature. It is planned that further analyses of worn Cu-Al surfaces will be performed before the end of CY 1990.

**Project Title:** Examination of Sapphire Substrate Surfaces (90-030) *J. J. Wert and J. E. Pawel*

User Center: MAUC

Status: Continuing

Our first training session on the Hitachi S-800 field emission scanning electron microscope in the HTML was completed on August 15, 1990. An as-deposited Fe (100 nm)/Al<sub>2</sub>O<sub>3</sub> specimen with five pull test sites was examined using a 2.5-kV electron beam. Even at this low voltage, however, the charging of the substrate in the regions where the film had been removed (the regions of interest in this investigation) obscured the details of the film failure sites, and means to overcome this experimental difficulty are still being studied.

**Virginia Polytechnic Institute & State University****Project Title:** Study of the Phase Transitions of Crystalline Aluminophosphates (89-054)*D. Young and M. J. Annen*

User Center: XRDUC

Status: Completed

In situ X-ray diffraction was used to study the transition of experimental aluminophosphates at elevated temperatures. In addition, changes in the hexagonal lattice of one of the materials designated as VPI-5 under various conditions of temperature, pressure, and humidity were determined.

**Project Title:** Measurement of Lattice Parameters as a Function of Temperature on Sodium Zirconium Phosphate (89-061) *S. M. Van Aken*

User Center: XRDUC and PPUC

Status: Completed

The high-temperature X-ray diffraction unit was used to conduct research on sodium zirconium phosphate. The information gathered will be used to develop new high-temperature thermal shock resistant materials for use in heat engines. Laser flash thermal diffusivity measurements have begun.

## APPENDIX A

## HTML PHYSICAL FACILITIES AND PERSONNEL

The Director of the HTML User Program is Dr. V. J. Tennery, who is also a section head in the Metals and Ceramics Division, parent organization of the HTML. Dr. Tennery is Chairman of the User Advisory Committee which is responsible for review of the research proposals from university and industrial users.

The administrative support staff and their functions are

Ms. Felicia Foust, User Coordinator  
Ms. Donna Conger, secretarial responsibilities, User Program  
Ms. Pam Rice, secretarial responsibilities, User Staff  
Ms. Robin Martin, secretarial assistance

A description of the function of each of the four user centers in the HTML is given here. The User Centers' staff are listed also.

## MATERIALS ANALYSIS USER CENTER (MAUC)

The materials characterization performed in the MAUC includes microstructural analyses and chemical and morphological characterization of surfaces utilizing sophisticated state-of-the-art instruments, generally not available to the user at the home institution.

The Group Leader of the MAUC is Mr. T. A. Nolan. Other staff and their equipment expertise are

Dr. L. F. (Larry) Allard, JEOL 4000EX (TEM) and JEOL 2000FX (AEM)  
Dr. A. (Ashok) Choudhury, VG ESCA/SIMSLAB2 and Auger  
Ms. D. W. (Dorothy) Coffey, Hitachi S-800 (FE-SEM) and sample preparation  
Mr. L. A. (Larry) Harris, VG ESCA/SIMSLAB2  
Mr. T. J. (Tommy) Henson, JEOL 733 Electron Microprobe  
Ms. K. L. (Karren) More, JEOL 2000FX (AEM) and JEOL 4000EX (TEM)  
Mr. T. A. (Tad) Dodson, computer services support

## MECHANICAL PROPERTIES USER CENTER (MPUC)

The MPUC is dedicated to the study of high-temperature mechanical properties of structural ceramics and alloys, including silicon carbides, silicon nitrides, aluminum oxide, transformation toughened zirconia, and high-temperature alloys such as superalloys. Facilities are available for the measurement of strength and toughness as a function of time, temperature, and stressing conditions. In addition to standard flexure strength measurements, the MPUC has 12 state-of-the-art tensile systems capable of evaluating the tensile strength of monolithic and ceramic matrix composites to temperatures up to 1600°C. New instrumentation includes the ability to study the tensile properties of single fibers to 1500°C and to directly measure the tensile deformation and fracture of continuous fiber-reinforced composites at high temperatures.

The Group Leader of the MPUC is Dr. M. K. Ferber. Other staff and their equipment expertise are

Dr. M. G. (Michael) Jenkins, tensile and flexure test facilities  
 Mr. V. T. (Tyler) Jenkins, ceramic specimen preparation  
 Mr. R. L. (Ralph) Martin, Universal test machines and sample preparation  
 Ms. R. L. (Becky) Freeny, Nanoindenter

#### PHYSICAL PROPERTIES (PPUC) AND X-RAY DIFFRACTION USER CENTERS (XRDUC)

The PPUC is organized to include instruments for determination of a broad range of the thermal physical properties of solid materials. Facilities are operational for the measurement of thermal diffusivity, specific heat, bulk thermal expansion, differential thermal analysis/thermal gravimetric analysis/mass spectra, and differential scanning calorimetry. Thermal diffusivity can be measured to 2000°C, and the other instruments operate to 1450°C. Efforts have begun to expand the modeling and measurement of thermal transport in materials.

X-ray diffraction is used to study anisotropic thermal expansion, phase equilibria, and microstructural defects of materials at either elevated temperatures or room temperature. The high-temperature X-ray diffraction furnace is capable of operation to 1600°C in air or other environments and to 2700°C in vacuum. Extensive data processing facilities and gas environment control capabilities are being developed.

The Group Leader of both the PPUC and the XRDUC is Dr. C. R. Hubbard. Other staff and their equipment expertise are

Mr. O. B. (Burl) Cavin, all X-ray equipment  
 Dr. R. B. (Ralph) Dinwiddie, laser flash diffusivity  
 Mr. W. D. (Wally) Porter, STA, DSC, Dilatometer

#### RESIDUAL STRESS USER CENTER

This new Center will be managed by Dr. C. R. Hubbard. It will provide sophisticated instruments for measurement of residual stress in and near the surface of ceramics and alloys. In many systems, these stresses are known to be critical to the reliability of the material in advanced applications. Specifications for a high flux X-ray generator and stress/texture goniometer system have been written and submitted for purchase. Plans are to have the X-ray part of this Center available to users by the latter part of FY 1991. A neutron diffraction residual stress facility is planned for a later time. It will be located at the High Flux Isotope Reactor and is planned for availability during the latter part of FY 1993, depending on the status of funding. The neutron facility will complement the X-ray facility by providing means for making stress measurements within the volume of advanced materials, and will be the first such dedicated facility in the United States.

## CERAMIC SPECIMEN PREPARATION USER CENTER

This new Center will be managed by Dr. M. K. Ferber. Specifications for the equipment have been submitted to purchasing. Some equipment is available and is located in two labs on the second floor in the core area. This center will provide an extensive capability for users to perform material finishing research plus conduct a number of highly accurate dimensional measurements with, for example, profilometers and high magnification comparators. Extensive computer control of the instruments is planned, including dimensional data acquisition and reduction. Two additional laboratory modules in the HTML were acquired for this new center.

## APPENDIX B

## LISTING OF THE ADVISORY COMMITTEES

## HTML Advisory Committee Members

	<u>Term Expires</u>
Dr. Maxine L. Savitz, Director Ceramic Components Division Garrett Processing Company 19800 South Van Ness Ave. Torrance, California 90509	1993
Mr. Woodie Howe Coors Technical Ceramic Company 1100 Commerce Park Drive Oak Ridge, Tennessee 37830	1992
Professor John J. Hren Head, Materials Science and Engineering North Carolina State University Box 7907, Yarborough Drive Raleigh, North Carolina 27695-7907	1992
Dr. Joseph N. Panzarino Norton Company Advanced Ceramics Division Goddard Road Northboro, Massachusetts 01532	1991
Dr. James W. Patten Director, Materials Engineering Cummins Engine Company 500 Jackson Street Columbus, Indiana 47201	1991

# HTML User Advisory Committee Members

Dr. V. J. Tennery, Director of the HTML, is permanent Chairman of this Committee.

	<u>Term Expires</u>
Dr. J. C. Danko, Director Center for Materials Processing The University of Tennessee 101 Perkins Hall Knoxville, Tennessee 37996-2000	1992
Mr. Lance Groseclose Allison Gas Turbine Operations General Motors Corporation Post Office Box 420 Indianapolis, Indiana 46206-0420	1992
Dr. Arvid Pasto GTE Laboratories, Inc. 40 Sylvan Road Waltham, Massachusetts 02254	1991
Ms. M. J. Rohr Oak Ridge Operations Office U.S. Department of Energy Oak Ridge, Tennessee 37831	1991
Dr. Linda Horton Metals and Ceramics Division Oak Ridge National Laboratory P. O. Box 2008 Oak Ridge, Tennessee 37831	1990

## APPENDIX C

## HTML CUMULATIVE USER EXPERIENCE

Figure C.1 illustrates the cumulative user days for industry, university, and local users in the HTML User Program for the entire 13 quarters of operation to date. Approximately 61% of the users have been local researchers, while about 25% have been from industry and 14% have been from universities.

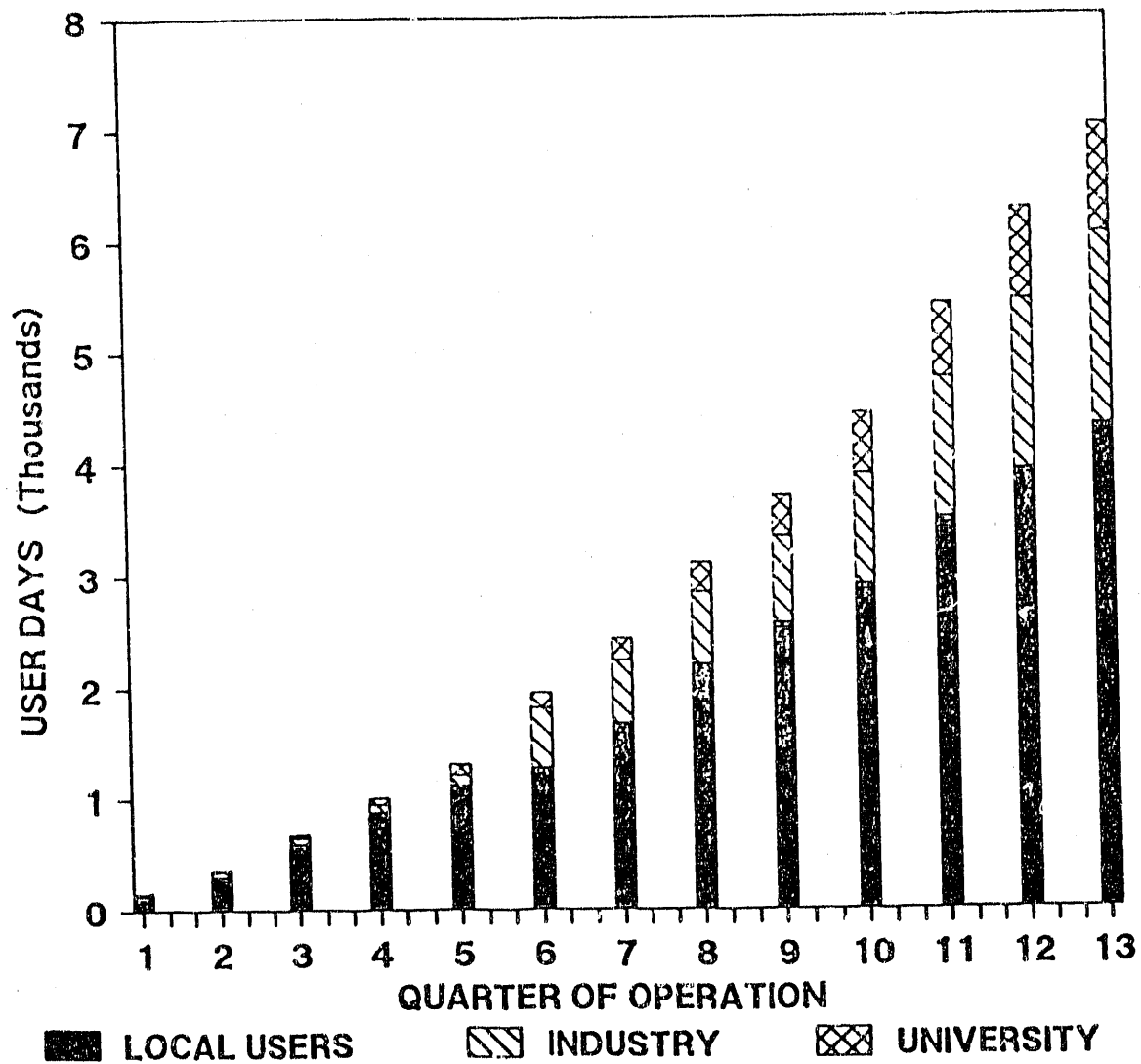


Fig. C.1. HTML cumulative user days from start of User Program.



## APPENDIX D

## PUBLICATIONS AND PRESENTATIONS

User Center Staff are indicated by an underline. The User's home institution is listed at the end of the citation. These publications are in the order of the name of the User Institution.

## INDUSTRY USERS PUBLICATIONS

K. R. Selkregg, K. L. More, S. G. Seshadri and C. H. McMurtry, "Microstructural Characterization of Silicon Nitride Ceramics Processed by Pressureless Sintering, Overpressure Sintering, and Sinter/HIP," Ceramic Engineering & Science Proceedings, L. G. Schioler, ed. 11 (7-8), p. 603 (1990), American Ceramic Society. CARBORUNDUM

M. V. Parish and J. D. Hodge, and C. R. Hubbard, "Thermal and Phase Analysis of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  Powder Compacts with Organic Acid Additives," *Proceedings of the Third International Conference on Ceramic Powder Processing Science*, San Diego, CA, February 4-7, 1990; (1990). CERAMICS PROCESS SUPERCONDUCTOR

Ran-Rong Lee and Wen-Cheng Wei, "Fabrication, Microstructure and Properties of SiC-AlN Ceramic Alloys," Ceramic Engineering & Science Proceedings, 11(7-8), p. 1094 (1990), American Ceramic Society. CERAMICS PROCESS SYSTEMS

R. T. Cassidy, W. E. Moddeman, L. F. Allard, "The Use of Austenitic Stainless Steels in Pyrotechnic Devices," *Journal of Metals*, in press. EG&G MOUND LABORATORIES

W. E. Moddeman, R. T. Cassidy, J. C. Birkbeck, and L. F. Allard, "TEM and Auger Examinations of the Oxide Film on an Al-Containing Austenitic Stainless Steel," *Journal of Metals*, in press. EG&G MOUND LABORATORIES

## INDUSTRY USERS PRESENTATIONS

T. A. Nolan, L. F. Allard, and M. H. Rawlins, "The Structure of Interfaces Resulting from Whisker Surface Pretreatments in SiC Whisker-Reinforced Ceramic Matrix Composites," MRS Fall Meeting 1989, Boston, MA, December 2-5, 1989. AMERICAN MATRIX, INC.

T. A. Nolan, L. F. Allard, D. W. Coffey, M. H. Rawlins, and R. W. Nixdorf, "Microstructural Characterization of a New VLS TiN Whisker Product," 14th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, FL, January 14-17, 1990. AMERICAN MATRIX, INC. First Prize in Poster Session

L. J. Klempner, J. D. Hodge, O. E. Cavin, and C. R. Hubbard, "Phase Formation of Yttrium Barium Cuprates," Third International Conference on Ceramic Powder Processing Science, San Diego, February 4-7, 1990. CERAMICS PROCESS SUPERCONDUCTOR

M. V. Parish and J. D. Hodge, and C. R. Hubbard, "Thermal and Phase Analysis of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  Powder Compacts with Organic Acid Additives," Third International Conference on Ceramic Powder Processing Science, San Diego, CA, February 4-7, 1990. CERAMICS PROCESS SUPERCONDUCTOR

J. D. Hodge and L. J. Klemptner, and C. R. Hubbard, "Phase Formation of Yttrium Barium Cuprates," annual meeting, American Ceramic Society, Dallas (1990). CERAMICS PROCESS SUPERCONDUCTOR

Ran-Rong Lee and Wen-Cheng Wei, "Fabrication, Microstructure and Properties of SiC-AlN Ceramic Alloys," 14<sup>th</sup> Annual Conference on Composites and Advanced Ceramics, January 14-17, 1990. CERAMICS PROCESS SYSTEMS

G. E. Potter, A. K. Knudsen, A. C. Choudhury, and J. C. Tou, "Characterization of Aluminum Nitride Materials with SIMS," annual meeting, American Ceramic Society, Dallas (1990). DOW CHEMICAL

W. E. Moddemar, R. T. Cassidy, L. F. Allard, "TEM and Auger Examinations of Oxidized Al-Containing Austenitic Stainless Steels for Glass/Metal Seal Applications," 14th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, FL, January 14-17, 1990. EG&G MOUND LABORATORIES

L. F. Allard, D. W. Coffey, W. E. Moddeman, and D. P. Kramer, "Nickel in LAS Glass to Metal Interfaces, Characterized by TEM and XPS," annual meeting, American Ceramic Society, Dallas (1990). EG&G MOUND LABORATORIES

L. F. Allard, D. W. Coffey, W. E. Moddeman, and D. P. Kramer, "Nickel in Las Glass to Metal Interfaces, Characterized by TEM and XPS," 14<sup>th</sup> Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, FL, January 14-17, 1990. EG&G MOUND LABORATORIES

Arvid Pasto, Ceramic Tensile Testing at GTE Laboratories, Inc.," presented at the Tensile Testing Meeting at Oak Ridge National Laboratory, March 5, 1990. GTE LABORATORIES

M. K. Ferber, T. A. Nolan, M. G. Jenkins, and R. Yeckley, "Fatigue and Creep Behavior of Silicon Nitride at Elevated Temperatures," annual meeting, American Ceramic Society, Dallas (1990). NORTON TRW

T. A. Nolan, L. F. Allard, D. W. Coffey, M. H. Rawlins, and R. W. Nixdorf, "Microstructural Characterization of VLS TiN Whiskers," annual meeting, American Ceramic Society, Dallas (1990). REMAXCO

## UNIVERSITY USERS PUBLICATIONS

Philip H. McCluskey, Robert K. Williams, Ron S. Graves, and Terry N. Tiegs, "Thermal Diffusivity/Conductivity of Alumina-Silicon Carbide Composites," *J. Am. Cer. Soc.*, Vol. 73, No. 2 (February 1990). ALFRED UNIVERSITY

James S. Wolf and O. Burl Cavin, "Oxidation and Sulfidation of Type 310S Stainless Steel at Elevated Temperatures," *Proceedings of the 29<sup>th</sup> Annual Conference of Metallography* "International Symposium on High Temperature Oxidation and Sulfidation Processes," Hamilton, Ontario, Canada, Aug. 26-30, 1990. CLEMSON UNIVERSITY

I. Baker, R. A. Padgett, and E. M. Schulson, "Auger Electron Spectroscopy Study of  $\text{Ni}_3\text{Si}$ ," *Scripta Met.*, Vol. 23, 1969-1974 (1989). DARTMOUTH COLLEGE

W. J. Lackey, J. A. Hanigofsky, M. J. Shapiro, W. B. Carter, D. N. Hill, E. K. Barefield, E. A. Judson, D. F. O'Brien, Y. S. Chung, T. S. Moss, and K. L. More, "Preparation of Superconducting Wire by Deposition of  $\text{YBa}_2\text{Cu}_3\text{O}_x$  Onto Fibers," *Proceedings of the 11<sup>th</sup> International Conference on Chemical Vapor Deposition*, Seattle, WA, October 14-19, 1990, Electrochemical Society. GEORGIA TECH

M. J. Shapiro, K. L. More, W. J. Lackey, J. A. Hanigofsky, D. N. Hill, W. B. Carter, E. K. Barefield, E. A. Judson, D. F. O'Brien, R. Patrick, and Y. S. Chung, "Interaction of Chemically Vapor Deposited  $\text{YBa}_2\text{Cu}_3\text{O}_x$  with Yttria Stabilized Zirconia Substrates," accepted for publication in *J. Amer. Cera. Soc.* GEORGIA TECH

J. A. Hanigofsky, K. L. More, W. J. Lackey, and W. Y. Lee, "Characterization of the BN + AlN Single Phase and Codeposition System," accepted for publication in the *J. Amer. Cera. Soc.* GEORGIA TECH

G. B. Freeman, W. Y. Lee, W. J. Lackey, J. A. Hanigofsky, and K. L. More, "Grain Structure and Growth of Dispersed Phase NB-AlN Coatings Grown Via Chemical Vapor Deposition," *Proceedings of the 1990 MRS Fall Meeting*, Boston, November 26-December 1, 1990. GEORGIA TECH

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E. L. Fuller, Jr., and V. J. Tennery, "Analytical Characterization of Coal Surfaces and Interfaces," Proceedings of the AR&TD Direct Utilization and Instrumentation and Diagnostics Contractors Review Meeting, Pittsburgh, September 16-18, 1990.

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L. F. Allard, T. A. Nolan, and T. N. Tiegs, "The Ultrastructure of SiC Whiskers: Crystallography and Chemistry of Surfaces and Inclusions," invited talk, TMS Meeting, Indianapolis, IN, October 1989.

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R. E. Clausing, L. Heatherly, E. D. Specht, and K. L. More, "Growth Mechanisms Film Morphology, Texture and Stresses for HFACVD Diamond Film Growth," NATO Meeting on Diamond and Diamondlike Films and Coatings, Pascoli, Italy, July 22-August 3, 1990.

R. E. Clausing, L. Heatherly, E. D. Specht, and K. L. More, "Texture Development in Diamond Films Grown by Hot Filament CVD Processes," 2nd International Conference on New Diamond Science and Technology, Washington, D.C., September 24-27, 1990.

R. A. Lowden, K. L. More, and R. D. James, "The Effects of Oxidation on the Properties of the Fiber-Matrix Interface in Nicalon/SiC Composites," Fossil Energy AR&TD Materials Program Conference Oak Ridge, TN, May 15-17, 1990.

D. P. Stinton, R. H. Krabill, K. L. More, D. M. Henmbree, and R. A. Lowden, "Characterization of Fiber-Reinforced SiC-Matrix Composites Fabricated by Chemical Vapor Infiltration," Fossil Energy AR&TD Materials Program Conference, Oak Ridge, TN, May 15-17, 1990.

R. A. Lowden, T. M. Besmann, and K. L. More, "The Effect of Molybdenum and Chromium Additions on the Oxidation Properties of CVD Silicon Carbide and Silicon Nitride," 14<sup>th</sup> Ann. Conf. Composites and Advanced Ceramics, Cocoa Beach, FL, January 14-17, 1990.

K. L. More, S. P. Withrow, and T. E. Haynes, "Growth of Epitaxial SiC Layers Onto On- and Off-Axis 6H-SiC Substrates by Ion Beam Deposition," MRS Fall Meeting, Boston, MA, November 27-December 2, 1989, MRS

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W. D. Porter, C. R. Hubbard, R. B. Dinwiddie, and O. B. Cavin, "Research Opportunities at the High Temperature Materials Laboratory Physical Properties and X-Ray Diffraction User Centers," 19<sup>th</sup> NATAS Conference, Cambridge, MA, September 23-26, 1990.

C. J. Sparks, W. D. Porter, J. H. Schneibel, W. C. Oliver, and C. G. Golec, "Formation of Cubic L<sub>1</sub><sub>2</sub> Phases from Al<sub>3</sub>Ti and Al<sub>3</sub>Zr by Transition Metal Substitution for Al," MRS Spring Meeting, San Francisco, CA, April 16-20, 1990.

V. J. Tennery, "The High Temperature Materials Laboratory: A New Experience at a National Facility," R&D Facilities Conference, New Concepts for Flexible, Cost-Effective Buildings, Philadelphia, PA, May 9-10, 1990.

V. J. Tennery and M. K. Ferber, "Cooperative International Program on Mechanical Strength Measurements of Ceramics," Annual Automotive Technology Development Contractors Coordination Meeting, Dearborn, MI, October 22-25, 1990.

V. J. Tennery and M. K. Ferber, "Fracture Strength Analysis of Silicon Nitride and Silicon Carbide Ceramics from an International Cooperative Research Program," Japan Fine Ceramics Association Headquarters, Tokyo, Japan, May 23, 1990.

V. J. Tennery, E. L. Fuller, Jr., T. A. Nolan, L. F. Allard, K. S. Ailey-Trent, D. W. Coffey, and L. A. Harris, "Analytical Characterization of Coal Surfaces and Interfaces," AR&TD Direct Utilization and Instrumentation and Diagnostics Contractors Review Meeting, Pittsburgh, PA, September 16-18, 1990.

## PENDING PRESENTATIONS

37<sup>th</sup> Sagamore Conference on Structural Ceramics, Plymouth, MA, October 1-4, 1990.

M. K. Ferber, M. G. Jenkins, and V. J. Tennery, "Comparison of Tension, Compression, and Flexure Creep for Alumina and Silicon Nitride Ceramics."

11<sup>th</sup> International Conference on Chemical Vapor Deposition, Seattle, WA, October 14-19, 1990

W. J. Lackey, J. A. Hanigofsky, M. J. Shapiro, W. B. Carter, D. N. Hill, E. K. Barefield, E. A. Judson, D. F. O'Brien, Y. S. Chung, T. S. Moss, and K. L. More, "Preparation of Superconducting Wire by Deposition of  $\text{YBa}_2\text{Cu}_3\text{O}_x$  Onto Fibers." GEORGIA TECH

Pacific Coast meeting of the American Ceramic Society, Seattle, WA, October 25-27, 1990.

M. G. Jenkins, M. K. Ferber, and J. A. Salem, "Increased Fracture Resistance Due to Beneficial Residual Stresses in a Particulate Ceramic Composite."

2nd International Ceramic Science and Technology Congress, Orlando, FL, November 12-15, 1990.

R. D. James, R. A. Lowden, and K. L. More, "The Effects of Oxidation and Combustion Environment on the Properties of Nicalon/SiC Composites."

K. L. More, D. A. Koester, and R. F. Davis, "Creep Behavior of a SiC Whisker-Reinforced  $\text{Si}_3\text{N}_4$  Composite in Air and Nitrogen."

Normand D. Corbin and Kerry N. Siebein, and R. A. Padgett, "Interfaces in SiC Whisker Reinforced  $\text{Si}_3\text{N}_4$  Composites." NORTON COMPANY

MRS 1990 Fall Meeting, Boston, November 26-December 1, 1990

W. J. Lackey, J. A. Hanigofsky, M. J. Shapiro, W. B. Carter, D. N. Hill, E. K. Barefield, E. A. Judson, D. F. O'Brien, Y. S. Chung, T. S. Moss, and K. L. More, "Preparation of Superconducting Wire by Deposition of  $\text{YBa}_2\text{Cu}_3\text{O}_x$  Onto Fibers." GEORGIA TECH

G. B. Freeman, W. Y. Lee, W. J. Lackey, J. A. Hanigofsky, and K. L. More, "Grain Structure and Growth of Dispersed Phase NB-AlN Coatings Grown Via Chemical Vapor Deposition." GEORGIA TECH

L. F. Allard, A. K. Datye, and R. T. Paine, "Atomic Structure of Crystalline Boron Nitride Interface with Ceramic Substrates." UNIVERSITY OF NEW MEXICO

C. R. Hubbard, R. B. Dinwiddie, W. D. Porter, and M. K. Ferber, "The Effect of Intergranular Phases and Thermal Treatment on the Thermophysical Properties of Silicon Nitride."



15th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, January 13-16, 1991.

W. S. Hong, J. M. Sater, and M. A. Rigdon, "The Effect of Varying Test Parameters on the Measured High Temperature Strength of SCS-6 Fiber." INSTITUTE FOR DEFENSE ANALYSIS

K. L. More, D. A. Koester, and R. F. Davis, "Creep of a SiC Whisker-Reinforced  $\text{Si}_3\text{N}_4$  Composite in Compression and Bending." NORTH CAROLINA STATE UNIVERSITY

R. E. Clausen, L. Heatherly, E. D. Specht, K. L. More, and L. L. Horton, "Growth Mechanisms, Film Morphology, and Defect Characterization for CVD Diamond Films."

E. Ballard, P. Predecki, C. R. Hubbard, "Residual Strains in  $\text{Al}_2\text{O}_3/\text{SiCw}$  Composite from 25-1000C,"

M. K. Ferber and T. N. Tiegs, "Effects of Post-Sintering Microwave Treatments Upon the Mechanical Performance of Silicon Nitride."

T. A. Nolan, M. K. Ferber, and D. W. Coffey, "Microstructural Characterization of Tensile and Flexural Creep Deformation and Fatigue in a  $\text{Si}_3\text{N}_4$  Ceramic."

## PATENT APPLICATIONS

A disclosure entitled "Microwave-Compatible Dilatometer," by H. K. Kimrey, Jr., M. A. Janney, and M. K. Ferber, ESID 652-X, was submitted on April 10, 1990.

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